APPENDIX G
QUALITY ASSURANCE/ QUALITY CONTROL (QA/QC) CONSIDERATIONS

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G.0 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) CONSIDERATIONS

G.1 Introduction

The following sections provide guidance for QA/QC. More detailed guidance, pertaining largely to physical and chemical evaluations, is provided in EPA (1995). This new QA document is applicable to both the Inland Testing Manual and to the Ocean Disposal "Green Book" (EPA/USACE, 1991), and will: 1) provide guidance on the development of QA project plans for ensuring the reliability of data gathered to evaluate dredged material proposed for discharge under the CWA or the MPRSA; 2) outline procedures that need to be followed when sampling and analyzing sediments, water, and tissues; and 3) provide recommended target detection limits (TDLs) for chemicals of concern.

A quality assurance (QA) program integrates management and technical practices into a single system to guarantee quality environmental data. The purpose of a QA program in a dredged material evaluation is to provide environmental data that are sufficient, appropriate, and of known and documented quality. Major elements of a QA program are:

- human resource training
- QA management plan (QAMP)/QA project plan (QAPP)
- management system reviews
- data quality objectives (DQOs)
- standard operating procedures (SOPs)
- project specific technical assessments.

QA project plans provide, in one place, a detailed plan for the activities performed at each stage of the dredged material evaluation (including appropriate sampling and analysis procedures) and outline project-specific data quality objectives that should be achieved for field observations and measurements, physical analyses, laboratory chemical analyses, and biological tests. Data quality objectives must be defined prior to initiating a project and adhered to for the duration of the project in order to guarantee acquisition of reliable data. This is accomplished by integrating quality control (QC) into all facets of the project, including development, implementation, and evaluation. QC is the routine application of procedures for determining bias and precision. QC procedures include activities such as preparation of replicate samples, spiked samples, blanks; calibration and standardization; sample custody and recordkeeping. Audits, reviews and compilation of complete and thorough documentation are activities used to verify compliance with pre-defined QC procedures. Through periodic reporting, these activities provide a means for management to track project progress and milestones, performance of measurement systems, and data quality.

A complete QA/QC effort for a dredged material testing program has two major components: a QA program implemented by the responsible governmental agency (the data user), and QC programs implemented by sampling and laboratory personnel performing the tests (the data generators). QA programs are also implemented by each field contractor and each laboratory. Typically, all field and laboratory data generators agree to adhere to the QA/QC of the data user for the contracted project as specified in the project QAPP. EPA (1987) provides useful guidance and may be followed on all points that are not in conflict with the guidance in this manual.

G.1.1 Government (Data User) Program

The USACE must implement a QA program to ensure that all program elements and testing activities (including field and laboratory operations) in the dredged material evaluation comply with the procedures in the QA project plan or with other specified guidelines for the production of environmental data of known quality. QA oversight is the responsibility of the USACE District Office, working in conjunction with the EPA Region. USACE Districts are responsible for ensuring that both the data submitted with permit applications, and that laboratories under contract to their Districts comply with the QA needs of the regulations and guidelines governing dredged material evaluations. The QA program should be designed with the assistance of programmatic and scientific expertise from both EPA and USACE. Other qualified sources of QA program management should be contacted as appropriate. Some specific QA considerations in contract laboratory selection are discussed by Sturgis (1990) and EPA (1991a).

G.1.2 Contractor (Data Generator) Program

Each office or laboratory participating in a dredged material evaluation is responsible for using procedures which assure that the accuracy (precision and bias), representativeness, comparability, and completeness of its data are known and documented. To ensure that this responsibility is met, each participating organization should have a project manager and a written QA management plan that describes, in specific terms, the management approach proposed to assure that each procedure under its direction complies with the criteria accepted by EPA and USACE. This plan should describe a QA policy, address the contents and application of specific QA project plans, and specify training requirements. All field measurements, sampling, and analytical components (physical, chemical, and biological) of the dredged material evaluation should be discussed.

For the completion of a dredged material testing project, the project manager of each participating organization should establish a well-structured QA program that ensures the following:

- development, implementation, and administration of appropriate QA planning documents for each study

- inclusion of routine QC procedures for assessing data quality in all field and laboratory standard operating procedures (SOPs)

- performance of sufficiently detailed audits at intervals frequent enough to ensure conformance with approved QA project plans and SOPs

- periodic evaluation of QC procedures to improve the quality of QA project plans and SOPs

- implementation of appropriate corrective actions in a timely manner.

G.2 The QA Project Plan

The QA project plan should be developed by the applicant or contractor for each dredged material evaluation, in accordance with EPA (1995). The QA project plan provides an overall plan and contains specific guidelines and procedures for the activities performed at each stage of the dredged material testing program, such as dredging site subdivision, sample collection, bioassessment procedures, chemical and physical analyses, data quality standards, data analysis and reporting. In particular, the QA plan addresses required QC checks, performance and system audits, QA reports to management, corrective actions, and assessment of data accuracy (precision and bias), representativeness, comparability and completeness. The plan should address the quantity of data required to allow confident and justifiable conclusions and decisions. QA project plans are particularly useful for work that involves many people or for projects that continue over a long period. When many people are involved, the plan ensures that everyone has a thorough understanding of the goals and procedures of the program. When work is conducted over a long period, the plan provides a basis for continuity, ensuring that procedures do not slowly change over time without the persons involved in the program evaluating the nature of the changes and their possible impact on data quality.

Each of the following items should be considered for inclusion in the QA Project Plan:

- Project description (G.2.1)
- QA organization; personnel responsibilities and qualifications (G.2.2)
- QA objectives for measurement data in terms of accuracy, representativeness, comparability, and completeness (G.2.3)

- Standard operating procedures (G.2.4)
- Sampling strategy and procedures (G.2.5)
- Sample custody and documentation (G.2.6)
- Calibration procedures (G.2.7)
- Analytical procedures (G.2.8)
- Data validation, reduction and reporting (G.2.9)
- Internal QC checks (G.2.10)
- Performance and system audits (G.2.11)
- Facilities (G.2.12)
- Preventative maintenance (G.2.13)
- Calculation of data quality indicators (G.2.14)
- Corrective actions (G.2.15)
- QA reports to management (G.2.16).

G.2.1 Project Description

A project description should be provided that defines project goals and illustrates how the project will be designed to obtain the information needed to achieve those goals. Sufficient detail and information should be included to allow decisions during the joint EPA and USACE review and the final USACE approval phases. Where appropriate, the following information should be included in this section of the QA project plan:

- objectives and scope of the project
- any historical information relevant to the dredging operation
- intended activities further described in flow diagrams, tables, and charts
- schedule of tasks and milestones
- intended use of acquired data.

G.2.2 QA Organization; Personnel Responsibilities and Qualifications

A clear delineation of the QA organization and line of authority is essential for the development, implementation, and administration of a QA program. This should include all technical personnel, including key individuals responsible for ensuring sufficient QC is being incorporated into the project. Organizational charts or tables should be used in the QA project plan to describe the management structure, personnel responsibilities, and the interaction among functional units. Each QA task should be fully described and the responsible individual and associated organization named. An example of a QA organization flow diagram is provided in Appendix G.4.

Technical staff are responsible for the validity and integrity of the data produced. The QA staff should be responsible for ensuring that all personnel performing tasks related to data quality are appropriately qualified. Records of qualifications and training of personnel should be kept current for verification by internal QA personnel or by EPA and USACE.

G.2.3 Data Quality Objectives

Data quality objectives are used to ensure that the data are acceptable. They define performance-based goals for accuracy (precision and bias), representativeness, comparability, and completeness as well as the required sensitivity of chemical measurements (i.e., target detection limits, TDLs). Accuracy is defined in terms of bias (how close the measured value is to the true value) and precision (how variable the measurements are when repeated). Data quality objectives should be based on the intended use of the data, technical feasibility, and consideration of cost. Numerical quality objectives should be summarized in a table, with all data calculated and reported in units consistent with other organizations reporting similar data, to allow comparability of data bases. All measurements should be made so that results are representative of the medium (e.g., water, sediments, tissue) being measured. Data quality objectives for precision and bias established for each measurement parameter should be based on prior knowledge of the measurement system employed, method validation studies, and the requirements of the specific project. An example of a data quality objectives summary for laboratory measurements is provided in Appendix G.4.

G.2.4 Standard Operating Procedures

Standard operating procedures (SOPs) are written descriptions of routine methods and should be provided for as many methods used during the dredged material evaluation as possible. A large number of field and laboratory operations can be standardized and presented as SOPs. Once these procedures are specified, they can be referenced or provided in an appendix of the QA project plan. Only modifications to SOPs or non-standard procedures need to be explained in the main body of the QA project plan (e.g., in the "sampling procedures" or "analytical procedures" section). General types of procedures benefiting from SOPs are field measurements ancillary to sample collection (e.g., depth of overlying water, sampling depth, water quality measurements, mixing model input measurements), chain-of-custody, sample handling and shipment, and routine analytical methods for chemical analyses. SOPs ensure that all persons conducting work are following the same procedures and that the procedures do not change over time. All personnel should be thoroughly familiar with the SOPs before work is initiated. Deviations from SOPs may affect data quality and integrity. If it is necessary to deviate from approved SOPs, these deviations must be documented and approved through an appropriate chain-of-command which may include USACE and EPA. Personnel responsible for ensuring the SOPs are adhered to must be identified in the QA Project Plan. Example SOPs are provided in Appendix D of EPA (1995).

G.2.5 Sampling Strategy and Procedures

A sampling strategy should be developed to ensure that the sampling design supports the planned data use. The sampling strategy will strongly affect the representativeness, comparability, and completeness that might be expected for field measurements. In addition, the strategy for collecting field QC samples (e.g., replicates) will assist in the determination of how well the total variability of a field measurement can be documented. Therefore, development of the sampling strategy should be closely coordinated with development of data quality objectives discussed in Section G.2.3.

To reduce sampling error, all methods, procedures, and equipment to be used in the field should be documented in a sampling plan which has been authorized and which is readily available to all personnel. The purpose of this sampling plan is to provide a blueprint for all field work by defining in detail the appropriate sampling and data collection methods (in accordance with the established data quality objectives). Written procedures or checklists for field equipment, sample container preparation, sample preservation, labelling and numbering systems, and shipping procedures must be appropriate. Methods to record and report deviations from the sampling plan must also be described. An alteration checklist form is generally appropriate to implement required changes. An example of such a checklist is provided in Appendix G.4.

G.2.6 Sample Custody and Documentation

Sample custody and documentation are vital components of all dredged material evaluations, particularly if any of the data may be used in a court of law. It is important to record all events associated with a sample so that the validity of the resulting data may be properly interpreted. Documentation is necessary during the field effort when samples are collected and in the laboratory where both chemical and biological analyses are performed. Thorough documentation provides a means to track samples from the field through the laboratory and prevent sample loss. The contents and location of all documents related to dredged sediment samples should be specified, and access to the samples should be controlled. Where samples may be needed for potential litigation, chain-of-custody procedures should be followed. Chain-of-custody procedures are initiated during sample collection. They include a descriptive label and tracking report forms for both the field and laboratory. An example of a label, field tracking report form, laboratory tracking report form and chain-of-custody record is provided in Appendix G.4.

G.2.6.1 Field Operations

The potential for sample deterioration and/or contamination exists during sample collection, handling, preservation, and storage. Approved protocols and SOPs should be followed to ensure all field equipment is acceptably calibrated and to prevent deterioration or contamination. Experienced personnel should be responsible for maintaining the sample integrity from collection through analysis. A complete record of all field procedures, an inventory log, and a tracking log should be maintained. A field tracking report should identify sample custody and conditions in the field prior to shipment.

Dates and times of collection, station locations, sampling methods, and sample handling, preservation, and storage procedures should be documented immediately, legibly, and indelibly so that they are easily traceable. Any circumstances potentially affecting sampling procedures should be documented. The data recorded should be thorough enough to allow station relocation and sample tracking. An example of a station location log is provided in Appendix G.4. Any field preparation of samples should also be described. Samples should be identified with a pre-prepared label containing at least the following information:

- project title
- sample identification number
- location (station number) and depth
- analysis or test to be performed
- preservation and storage method
- date and time of collection
- special remarks if appropriate
- initials of person collecting the sample
- name of company performing the work.

G.2.6.2 Laboratory Operations

The responsible party who will act as sample custodian at the laboratory facility should be identified. This individual has authority to sign for incoming field samples and has the responsibility to obtain documents of shipment and verify the data entered on the sample custody records. A laboratory-tracking report should be prepared for each sample. The location of samples processed through chain-of-custody must be known at all times. Samples to be used in a court of law must be stored in a locked facility to prevent tampering or alteration.

A procedure should be established for the retention of all appropriate field and laboratory records and samples as various tasks or phases are completed. Replicates, subsamples of analyzed samples, or extra unanalyzed samples should be kept in a storage bank. These samples can be used to scrutinize anomalous results or for supplemental analyses, if additional information is needed. All samples should be properly stored and inventoried. The retention and archiving procedure should indicate the storage requirements, location, indexing codes, retention time, and security requirements for samples and data.

G.2.7 Calibration Procedures

Calibration procedures should be included for each instrument used during the study. The appropriate procedures used to assure that field and laboratory equipment are functioning properly should be documented in this section. This information can be provided in tabular format. The planned frequency for recalibration should be provided as well as a list of the calibrations standards to be used and their sources, including traceability procedures. Instrumentation that requires routine calibration includes, for example, navigation devices, analytical balances, and water quality meters.

G.2.8 Analytical Procedures

The methods cited in the analytical procedures section of a QA project plan are used to meet the data quality objectives for a dredged material evaluation. (Section 9 of this Manual provides guidance on the selection of physical and chemical analyses to aid in evaluating dredged material proposed for disposal, and on the methods used to analyze these parameters.) In all cases, proven, state-of-the-art methods should be used. Sample analysis procedures are identified in this section of the QA project plan by reference to established, standard methods. Any modifications to established, standard methods and any specialized, nonstandard procedures should be described in detail in this section of the plan.

G.2.9 Data Validation, Reduction and Reporting

Data validation involves all procedures used to accept or reject data after collection and prior to use. These include screening, editing, verifying, and reviewing through external performance evaluation audits. Data validation procedures ensure that objectives for data precision and bias were met, that data were generated in accordance with the QA project plan and SOPs, and that data are traceable and defensible. All data should be reported with their associated analytical sensitivity, precision, and bias. In addition, the level of quantification achieved by the laboratory should be compared to specific target detection limits. The following information should be included in the QA project plan:

- the principal criteria that will be used to validate data integrity during their collection and reporting

- the data reduction scheme planned for collected data including all equations used to calculate the concentration or value of the measured parameter and reporting units

- the methods used to identify and treat outliers and nondetectable data

- the data flow or reporting scheme from collection of raw data through storage of validated concentrations (a flowchart is usually necessary)

- statistical formulae and sample calculations planned for collected data

- key individuals who will handle the data in this reporting scheme.

QC procedures designed to eliminate errors during the mathematical and/or statistical reduction of data should also be included in the QA project plan. Quality control in data processing may include both manual and automated review. Input data should be checked and verified to confirm compatibility and to flag "outliers" for confirmation. Computerized data plots can be routinely used as a tool for rapid identification of outliers that can then be verified using standard analytical procedures.

Data entries should be dated when entered, and signed or initialled by the person making the measurement and the person entering the data. Changes to entries should be made so as not to obscure the original entry. They should indicate the reason for the change, the person making the change, and the date of change. In computer-driven data collection systems, the person responsible for direct data input should be identified at the time of input.

The data and information collected during the Tier I evaluation should be carefully reviewed as to their relevancy, completeness, and quality. The data must be relevant to the overall objective of the project, even though the objectives for these studies were different.

G.2.10 Internal Quality Control Checks

The various control samples that will be used internally by the laboratory or sample collection team to assess quality are described in this section of the QA project plan. For most environmental investigations, 10-30 percent of all samples may be analyzed specifically for purposes of quality control. In some special cases (e.g., when the number of samples is small and the need to establish the validity of analytical data is large), as many as 50 percent of all samples are used for this purpose. These QC samples may be used to check the bias and precision of the overall analytical system and to evaluate the performances of individual analytical instruments or the technicians that operate them. The most widely used QC samples are summarized in EPA (1995) and are as follows:

- blanks
- matrix spike samples
- surrogate spike compounds
- check standards, including:
  - spiked method blanks
  - laboratory control samples
  - reference materials
- matrix replicates (split in the laboratory from one field sample)
- field replicates (collected as separate field samples from one location).

The following sections discuss quality control procedures for sediment, water, and tissue analyses (see EPA, 1995 for further detail), as well as for biological analyses.

The USACE District or management authority for the program may require that certain samples be submitted on a routine basis to government laboratories for analysis, and EPA or USACE may participate in some studies. These activities provide an independent quality assurance check on activities being performed and on data being generated and are discussed in Section G.2.11.

G.2.10.1 Quality Control Considerations for Physical Analysis of Sediments

The procedures used for the physical analysis of sediments must include a QC component. QC procedures for grain-size analysis and total solids/specific gravity determinations are necessary to ensure that the data meet acceptable criteria for precision and bias. To measure precision, triplicate analyses should be performed for every 20 samples analyzed. TOC is a special case, where all samples should be analyzed in triplicate. In addition, one procedural blank per 20 samples should be run, and the results reported for TOC analysis. Standards used for TOC determinations must be verified by independent check standards to confirm the bias of the results. QC limits should be agreed upon for each analytical procedure, and should be consistent with the overall QA project plan.

G.2.10.2 Quality Control Considerations for Chemical Analysis of Sediments

Methods for the chemical analysis of contaminants of concern in sediments must include detailed procedures and requirements which should be followed rigorously throughout the evaluation. General procedures include the analysis of a procedural blank, a matrix duplicate, a matrix spike along with every 10 - 20 samples processed, and surrogate spike compounds (for organic analyses only). All analytical instruments should be calibrated at least daily. All calibration data should be submitted to the laboratory project QA coordinator for review. The QA/QC program must document the ability of the selected methods to address the high salt content of sediments from marine and estuarine areas.

Analytical precision can be measured by analyzing one sample in duplicate or triplicate for every 10 - 20 samples analyzed. If duplicates are analyzed, the relative percent difference should be reported. However, if triplicates are analyzed, the percent relative standard deviation should be reported.

G.2.10.3 Quality Control Considerations for Chemical Analysis of Water

Methods recommended for the chemical analysis of contaminants of concern in water include detailed QC procedures and requirements which should be followed closely throughout the evaluations. General procedures should include the analysis of a procedural blank, a matrix duplicate, a matrix spike for every 10 - 20 samples processed, and surrogate spike compounds (for organic analysis only). Analytical precision can be measured by analyzing one sample in triplicate or duplicate for every 10 - 20 samples analyzed. If duplicates are analyzed, the relative percent difference should be reported. However, if triplicates are analyzed, the percent relative standard deviation should be reported. Analytical bias can be measured by analyzing standard reference materials (SRMs), a matrix containing a known amount of a pure reagent. Recoveries of surrogate spikes and matrix spikes should be used to measure for precision and bias; results from these analyses should be well documented. Special QC is required for ICP and GC/MS analyses. Initial calibrations using three or five standards (varying concentrations) are required before analyzing samples. Subsequent calibration checks should be performed for every 10 - 20 samples analyzed.

G.2.10.4 Quality Control Considerations for Chemical Analysis of Tissue

As with sediments and water, methods recommended for the chemical analysis of contaminants of concern in tissues include detailed QC procedures and requirements which should be followed closely throughout the evaluations. General procedures should include the analysis of a procedural blank, a matrix duplicate, a matrix spike for every 10 - 20 samples processed, and surrogate spike compounds (for organic analyses only). Analytical precision can be measured by analyzing one sample in triplicate or duplicate for every 10 - 20 samples analyzed. If duplicates are analyzed, the relative percent difference should be reported. However, if triplicates are analyzed, the percent relative standard deviation should be reported. Analytical bias can be measured with the appropriate SRMs. Precision and bias determinations should be performed with the same frequency as the blanks and matrix spikes.

G.2.10.5 Quality Control Considerations for Biological Analyses

Quality controls for tests of biological effects and bioaccumulation must address all activities that affect the quality of the data (e.g., see EPA, 1991b). These activities include:

- source and condition of test organisms
- use of negative (non-toxic) and positive (reference toxicants) controls
- acceptability of test results and data evaluation.

Standard laboratory procedures must be followed in all testing including maintenance/measurement of environmental (e.g., water) quality conditions and blind testing.

G.2.10.5.1 Source and Condition of Test Organisms

Test organisms should be positively identified to species by qualified experts. Test organisms should appear healthy, behave normally, feed well, and have low mortality in cultures, during holding, and in test controls. The quality of test organisms from outside sources as well as those maintained in-house must be verified by conducting a reference toxicant test concurrently with the dredged material toxicity tests. The supplier should provide data with the shipment describing the history of the sensitivity of organisms from the same source culture, determined in monthly tests using suitable reference toxicants.

G.2.10.5.2 Reference Toxicants

Biological QC includes periodic reference toxicant tests with all stocks of organisms to be used in testing to determine the relative health of the organisms. The application and benefits of reference toxicant tests are discussed by Lee (1980). Detailed assistance in establishing a biological QC program can be provided by scientists from EPA or USACE.

Reference toxicants are routinely used to evaluate species sensitivity, laboratory performance and both intra- and inter- laboratory precision. The following chemicals provide good endpoints for a variety of species: freshwater species - sodium chloride, copper sulfate, potassium chloride, cadmium chloride, sodium dodecyl sulfate, diazinon; saltwater species - copper sulfate, cadmium chloride, sodium dodecyl sulfate, diazinon. It is required that a set of the above chemicals with difference modes of toxic action be used as reference toxicants in establishing comparative sensitivity between recommended species listed in Tables 11, 12 and 13 of the Manual and a species proposed as a substitute regional test species.

Reference toxicant tests should be performed routinely on all groups of organisms used in dredged material toxicity and bioaccumulation studies in order to determine their relative health and vigor. A single reference toxicant can be used to assess this in routine testing. Many chemicals may be used satisfactorily as reference toxicants (e.g., Lee, 1980; Wang, 1987; EPA, 1990, 1991c). Reference toxicant tests are performed in the absence of sediment and generally under static conditions. Water-only reference toxicant tests with benthic species may require some modification to "standard" test conditions used for pelagic species. A short term response to a standardized exposure is used as an indication of the relative health of the organisms. A geometric dilution series of five unreplicated concentrations is used plus a negative (dilution-water only) control. Although nominal concentrations are usually sufficient for reference toxicant tests, concentrations should be measured whenever possible. The concentration range should be selected to give greater than 50% mortality in at least one concentration and less than 50% mortality in at least one concentration. An initial range-finding test using a very wide range of concentrations may be necessary to determine the proper concentration range for reference toxicant tests. For each species, mortality is determined and the LC_{50} or EC_{50} is calculated as described in Appendix D.

A control chart should be developed for each reference toxicant/test organism combination used (e.g., see EPA, 1990). The LC_{50} or EC_{50} for each combination should be determined on a regular basis, and each combination tracked on a separate Average Control Chart (Figure G.1). Successive toxicity values should be plotted and examined to determine if the results are within the established limits. Commonly used limits are the mean±2 standard deviations. A minimum of five data points are necessary to develop the first set of limits. These limits are recalculated for each successive data point, until the statistics stabilize. Organisms are suitable for dredged material testing if results of reference toxicant testing fall within these limits. Outliers, or data which fall outside the upper and lower limits, suggest an atypical population. It is inappropriate to use that group of organisms for dredged material testing as the sensitivity of the organisms and the overall credibility of the test system would be suspect. Reference toxicant tests should be conducted at least monthly on each species cultured in-house, and should be performed on each lot of purchased or field-collected organisms. The basic concept and application of reference toxicant tests is discussed by Lee (1980). When sufficient reference toxicant data have been generated for a particular species, it may be possible to specify an acceptable LC_{50} or EC_{50} range for that species with that reference toxicant.

G.2.10.5.3 Acceptability of Test Results and Data Evaluation

For the test results to be acceptable, mean control survival must be ≥ 90% or the appropriate value for a particular test and end-point. If mean mortality is greater than 10% or the appropriate value for a particular test or endpoint in the control treatment for a particular test species, the test should be rejected and repeated. Unacceptable control mortality indicates that the organisms are being affected by stress other than contamination in the material being tested. Such stress may be due to injury or disease, unfavorable physical or chemical conditions in the test containers, improper handling or acclimation or possibly unsuitable or contaminated water. The potential effects of these and other variables should be carefully examined if the test is repeated.

An individual test may be conditionally acceptable if temperature, DO, and other specified conditions fall outside specifications. This depends on the degree of the departure and the objectives of the tests. The acceptability of the test will depend on the experience and professional judgment of the laboratory analyst and the reviewing staff of the regulatory authority. Any deviation from test specifications must be noted when reporting data from a test.

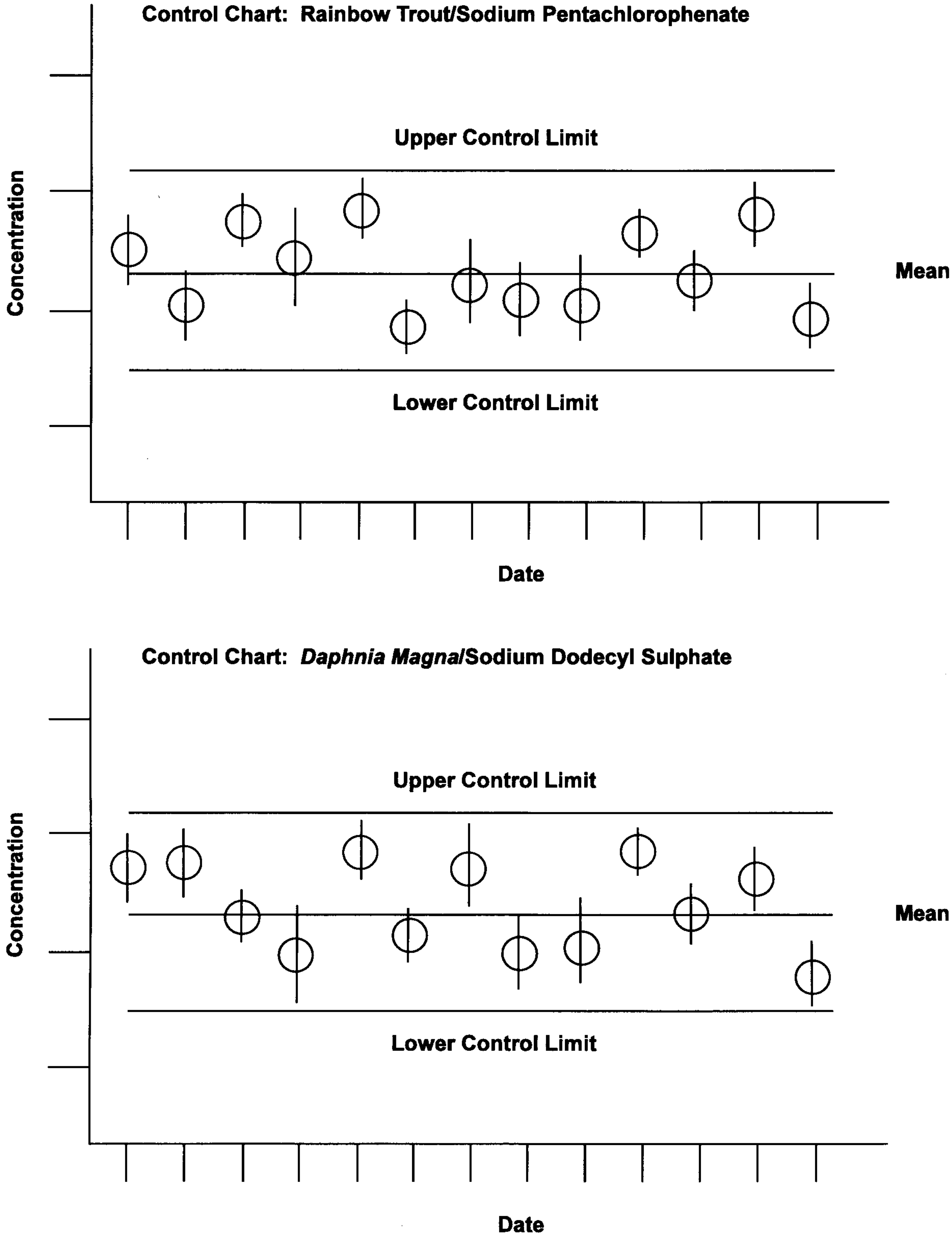


Figure G.1 Example Control Charts for Reference Toxicants.

G.2.11 Performance and System Audits

Audits include a careful evaluation of both field and laboratory QC procedures. They are an essential part of the field and laboratory QA program and consist of two basic types: performance audits and system audits. For example, analyses of performance evaluation samples may simply be used for comparison with the results of independent laboratories (a form of performance audit), or comprehensive audits may be conducted by the government of the entire field or laboratory operations (a system audit).

Performance and system audits should be conducted by individuals not directly involved in the measurement process. A performance auditor independently collects data using performance evaluation samples, field blanks, trip blanks, duplicate samples, and spiked samples. Performance audits may be conducted soon after the measurement systems begin generating data. They may be repeated periodically as required by task needs, duration, and cost. EPA (1991b) should be reviewed for auditing the performance of laboratories performing aquatic toxicity tests.

A systems audit consists of a review of the total data production process. It includes on-site reviews of field and laboratory operational systems. EPA and/or USACE will develop and conduct external system audits based on the approved project plan. An example of a systems audit checklist is provided in EPA (1995).

G.2.11.1 Pre-award Inspections

The pre-award inspection is a type of system audit for assessing the laboratory's overall capabilities. This assessment includes a determination that the laboratory personnel are appropriately qualified and that the required equipment is available and is adequately maintained. It establishes the groundwork necessary to ensure that tests will be conducted properly, provides the initial contact between government and laboratory staff, and emphasizes the importance that government places on quality work and products. The purpose of the pre-award inspection is to verify the following:

- The laboratory has an independent QA/QC program.
- Written work plans are available for each test that describe the approach to be used in storing, handling, and analyzing samples.
- Technically sound, written standard operating procedures (SOPs) are available for all study activities.

- Qualifications and training of staff are appropriate and documented.
- Approved analytical procedures are being followed.

G.2.11.2 Interlaboratory Comparisons (Chemical Analytical Laboratories)

It is important that data collected and processed at various laboratories be comparable. As part of the performance audit process, laboratories may be required to participate in analysis of performance evaluation samples related to specific projects. In particular, laboratory proficiency testing is recommended. Laboratory proficiency must be demonstrated before a laboratory negotiates a contract and yearly thereafter. Each laboratory participating in a proficiency test is required to analyze samples prepared to a known concentration. Analytes used in preparation of the samples must originate from a recognized source of standard reference material (SRM), such as the National Institute for Standards and Technology (NIST). Proficiency testing programs already established by either EPA or the USACE may be used, or a program may be designed specifically for dredged material evaluations. Analytical results are compared with predetermined criteria of acceptability.

In addition, the performance evaluation samples prepared by EPA Environmental Monitoring and Systems Laboratory (Las Vegas, Nevada) for the Contracts Laboratory Program (CLP) may be used to assess interlaboratory comparability. Analytical results are compared with predetermined criteria of acceptability (e.g., values that fall within the 95 percent confidence interval are considered acceptable). The QA project plan should indicate, where applicable, scheduled participation in all interlaboratory calibration exercises.

Reference materials are substances with well-characterized properties that are useful for assessing the bias of an analysis and auditing analytical performances among laboratories. SRMs are certified reference materials containing precise concentrations of chemicals, accurately determined by a variety of technically valid procedures, and are issued by the National Institute of Standards and Technology. Currently, SRMs are not available for the physical measurements of all contaminants in sediments; however, where possible, available SRMs or other regional reference materials that have been repeatedly tested should be analyzed with every 20 samples processed.

SRMs for most organic compounds are not currently available for seawater, but reference materials for many inorganic chemicals may be obtained from the organizations listed in Table G.1. Seawater matrix spikes of target analytes (e.g., seawater spiked with National Institute for Standards and Technology SRM 1647 for PAH) should be used to check analytical bias. Some available SRMs for priority pollutant metals in seawater are National Research Council of Canada seawater CASS-1 and seawater NASS-2.

Table G.1 Sources of Standard Reference Materials

PCBs		
National Research Council of Canada	Marine sediment	HS-1 and HS-2
PAHs		
National Research Council of Canada	Marine sediment	HS-3, HS-4, HS-5, HS-6
National Institute for Standards and Technology	Sediment	SRM #1647 and SRM #1597
Metals		
National Bureau of Standards	Estuarine sediment	SRM #1646
National Research Council of Canada	Marine sediment	MESS-1, BCSS-1, PACS-1
	Dogfish liver	DOLT-1
	Dogfish muscle	DORM-1
	Lobster hepatopancreas	TORT-1
International Atomic Energy Agency	Marine sediment	SD-N-1/2(TM)
	Fish flesh	MA-A-2(TM)
	Mussel tissue	MAL-1(TM)

Standard reference materials (SRMs) may be obtained from the following organizations:

Organic Constituents

U.S. Department of Commerce
National Institute for Standards of Technology
Office of Standard Reference Materials
Room B3111 Chemistry Building
Gaithersburg, Maryland 20899
Telephone: (301) 975-6776

Marine Analytical Chemistry Standards Program
National Research Council of Canada
Atlantic Research Laboratory
1411 Oxford Street
Halifax, Nova Scotia, Canada B3H 3Z1
Telephone: (902) 426-8280

Inorganic Constituents

U.S. Department of Commerce
National Institute for Standards and Technology
Office of Standard Reference Materials
Room B3111 Chemistry Building
Gaithersburg, Maryland 20899
Telephone: (301) 975-6776

Marine Analytical Chemistry Standards Program
National Research Council of Canada
Division of Chemistry
Montreal Road
Ottawa, Ontario, Canada K1A 0R9
Telephone: (613) 993-2359

SRMs for organic priority pollutants in tissues are currently not available. The National Institute of Standards and Technology is presently developing SRMs for organic analytes. Tissue matrix spikes of target analytes should be used to fulfill analytical accuracy requirements for organic analyses.

Because new SRMs appear constantly, current listings of appropriate agencies should be consulted frequently. SRMs that are readily available and commonly used are included in Table G.1.

G.2.11.3 Routine Inspections

Routine system audits during the technical evaluation ensure that laboratories are complying with the QA project plan. It is suggested that checklists be developed for reviewing training records, equipment specifications, QC procedures for analytical tasks, management organization, etc. An example of a systems audit is provided in EPA (1995). Districts should also establish laboratory review files for quick assessment of the laboratory's activity on a study, and to aid in monitoring the overall quality of the work. Procedures for external systems audits by the Districts are similar to the internal systems audits conducted by the laboratories themselves.

G.2.12 Facilities

The QA Project Plan should provide a complete, detailed description of the physical layout of the laboratory, define space for each test area, describe traffic-flow patterns, and document special laboratory needs. The design and layout of laboratory facilities are important to maintain sample integrity and prevent cross-contamination. The specific areas to be used for the various evaluations should be identified. Aspects of the dredging study that warrant separate facilities include the following:

- receiving
- sample storage
- sample preparation
- sample testing
- reagent storage
- data reduction and analysis.

G.2.13 Preventive Maintenance

The QA project plan should describe how field and laboratory equipment essential to sample collection and analysis will be maintained in proper working order. Preventive maintenance may be in the form of: 1) scheduled maintenance activities to minimize costly downtime and ensure accuracy of measurement systems, and 2) available spare parts, backup systems, and equipment. Equipment should be subject to regular inspection and preventive maintenance procedures to ensure proper working order. Instruments should have periodic calibration and preventive maintenance performed by qualified technical personnel, and a permanent record kept of calibrations, problems diagnosed, and corrective actions applied. An acceptance testing program for key materials used in the performance of environmental measurements (chemical and biological materials) should be applied prior to their use.

G.2.14 Calculation of Data Quality Indicators

The calculations and equations used routinely in QA review (e.g., relative percent difference of duplicates) as well as the type of samples (e.g., blanks, replicates) analyzed to assess precision, bias, and completeness of the data must be presented in the QA project plan. Routine procedures for measuring precision and bias include use of replicate analyses, standard reference materials, and matrix spikes. Completeness can be measured for each set of data received by dividing the number of valid (i.e., accepted) measurements actually obtained by the number of measurements that were planned.

G.2.15 Corrective Actions (Management of Nonconformance Events)

One purpose of any QA program is to identify nonconformance as quickly as possible. A nonconformance event is defined as any event that does not follow defined methods, procedures, or protocols, or any occurrence that may affect the quality of the data or study. A QA program should have a corrective action plan and should provide feedback to appropriate management authority defining how all nonconformance events were addressed and corrected.

Corrective actions fall into two categories: 1) handling of analytical or equipment malfunctions, and 2) handling of nonconformance or noncompliance with the QA requirements that have been established. During field and laboratory operations, the supervisor is responsible for correcting equipment malfunctions. All corrective measures taken must be documented and, if required, an alteration checklist must be completed.

Corrective action procedures must be described for each project and include the following elements:

- procedures for corrective actions when predetermined limits for data acceptability are exceeded (see "data quality objective" discussion in Section G.2.3)

- for each measurement system, identify the individual responsible for initiating the corrective action and also the individual responsible for approving the corrective action.

Corrective actions may be initiated as a result of other QA activities including performance audits, system audits, interlaboratory/interfield comparison studies, and QA program audits. An example of a corrective actions checklist is provided in Appendix G.4.

G.2.16 QA Reports to Management

QA Project Plans provide a mechanism for periodic reporting to management on the performance of measurement systems and data quality. At a minimum, these reports should include:

- periodic assessment of measurement data accuracy (precision and bias), and completeness

- results of performance and system audits

- significant QA problems and recommended solutions.

The individuals responsible for preparing the periodic reports should be identified. The final report for each project must include a separate QA section which summarizes data quality information contained in the periodic reports.

G.3 REFERENCES

EPA. 1987. *Quality Assurance/Quality Control (QA/QC) for 301(h) Monitoring Programs: Guidance on Field and Laboratory Methods.* EPA 430/9-86-004. Prepared by Tetra Tech, Inc., Bellevue WA, for the U.S. Environmental Protection Agency Office of Marine and Estuarine Protection. NTIS Number PB87-221164.

EPA. 1990. *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms.* EPA/600/4-90/027. U.S. Environmental Protection Agency, Office of Research and Development, Washington, D.C. 293 pp.

EPA. 1991a. *A Project Manager's Guide to Requesting and Evaluating Chemical Analyses.* Prepared by PTI Environmental Services, Bellevue, WA, for the U.S. Environmental Protection Agency Region 10, Puget Sound Estuary Program, Seattle, WA.

EPA. 1991b. *Manual for the Evaluation of Laboratories Performing Aquatic Toxicity Tests.* EPA/600/4-90/031. U.S. Environmental Protection Agency, Office of Research and Development, Washington, D.C.

EPA. 1991c. *Technical Support Document for Water Quality-Based Toxics Control.* EPA/505/2-90-001. U.S. Environmental Protection Agency, Office of Water, Washington, D.C.

EPA. 1995. *QA/QC Guidance for Sampling and Analysis of Sediments, Water, and Tissues for Dredged Material Evaluations. Phase I - Chemical Evaluations.* EPA 823-8-95-001. U.S. Environmental Protection Agency, Office of Water, Washington, D.C.

EPA/USACE. 1991. *Evaluation of Dredged Material Proposed for Ocean Disposal - Testing Manual.* EPA-503/8-91/001, Washington, DC.

Lee, D.R. 1980. Reference toxicants in quality control of aquatic bioassays. Pp. 188-199 In: A.L. Buikema, Jr. and J. Cairns, Jr. (Eds.), *Aquatic Invertebrate Bioassays.* ASTM STP 715. American Society for Testing and Materials, Philadelphia, PA.

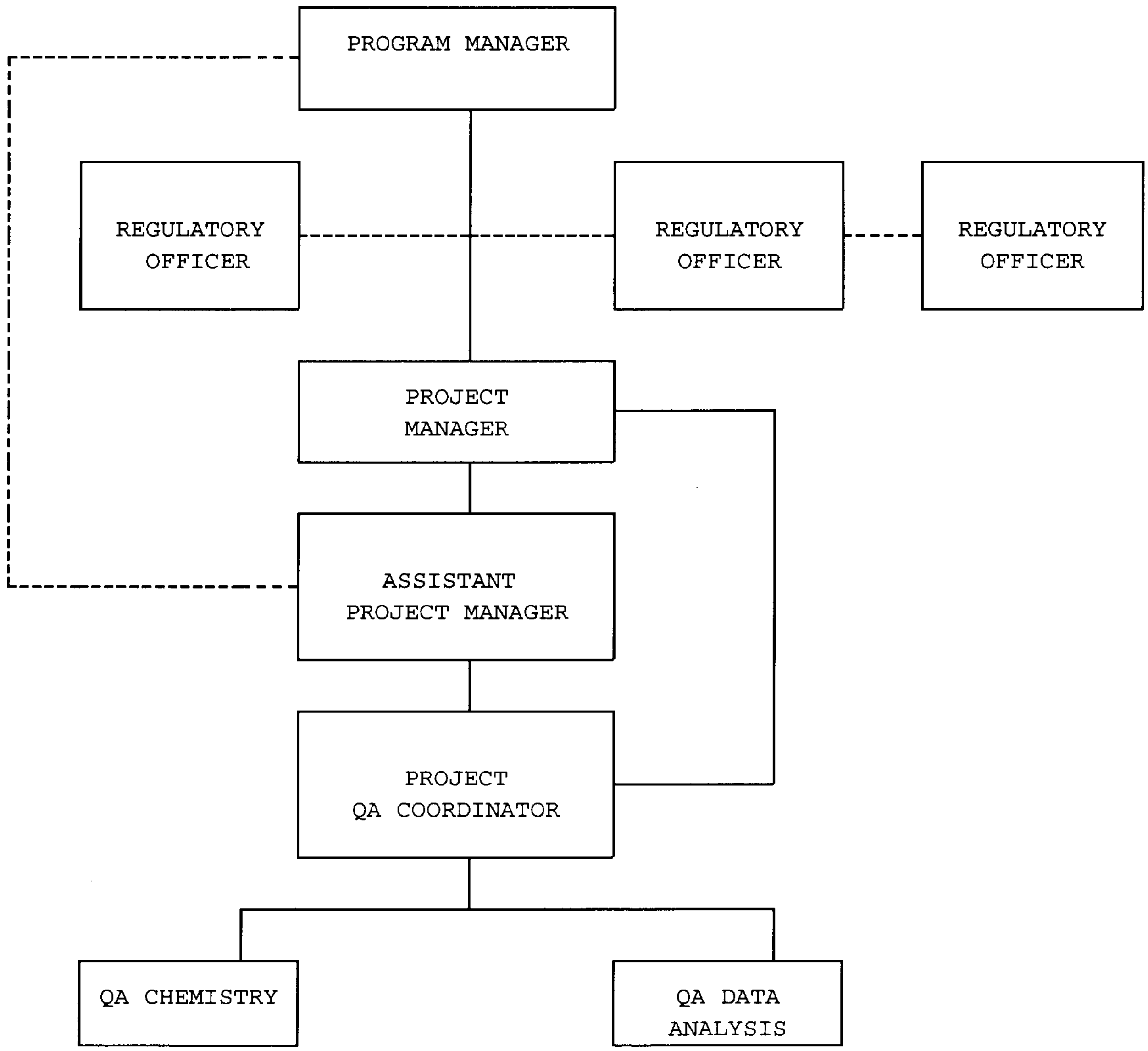
Sturgis, T.C. 1990. Guidance for contracting biological and chemical evaluations of dredged material. Tech. Rept. D-90-XX, U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS.

Wang, W. 1987. Chromate ion as a reference toxicant for aquatic phytotoxicity tests. Environ. Toxicol. Chem. 6: 955-960.

APPENDIX G.4
EXAMPLE QA/QC CHECKLISTS, FORMS, AND RECORDS

TABLE OF CONTENTS

QA PROGRAM ORGANIZATION FLOW DIAGRAM



EXAMPLE DATA QUALITY OBJECTIVES FOR PRECISION, ACCURACY, AND COMPLETENESS

Variable	Matrix	Units	Lower Limit of Detection	Accuracy	Precision (%)	Completeness (%)	Method	Reference	Maximum Holding Time
Volatiles	Sediment	µg/kg	5	*	±30%	99%	Purge & Trap/ GC-MS	EPA abc/x-cc-yyy (1975)	10 days
Grain Size	Sediment	Percent	0.01	--	±5%	99%	Sieve & pipet		Undetermined

ALTERATION CHECKLIST

Sample Program Identification: ______________________________

Material to be Sampled: ______________________________

Measurement Parameter: ______________________________

Standard Procedure for Analysis: ______________________________

____

Reference: ______________________________

____

____

Variation from Standard Procedure: ______________________________

____

____

Reason for Variation: ______________________________

____

____

Resultant Change in Field Sampling Procedure: ______________________________

____

____

Special Equipment, Material, or Personnel Required: ______________________________

____

____

Author's Name: ____________________ Date: ____________________

Approval: ____________________ Title: ____________________

Date: ____________________

GENERAL SAMPLE LABEL

(NAME OF SAMPLING ORGANIZATION)

PROJECT: ____________________

DATE: ____________________

TIME: ____________________

SAMPLE ID NO.: ____________________

MEDIA: ____________________

STATION NUMBER: ____________________

DEPTH: ____________________

PRESERVATION: ____________________

ANALYSES TO BE PERFORMED: ____________________

SAMPLED BY: ____________________

LAB NO.: ____________________

REMARKS: ____________________

FIELD TRACKING REPORT FORM

W/O No. ______________ Page __________

FIELD TRACKING REPORT: _______-_______
(LOC-SN)

FIELD SAMPLE CODE (FSC)	BRIEF DESCRIPTION	DATE	TIME	SAMPLER

LABORATORY TRACKING REPORT FORM

W/O No. ______________ Page __________

LABORATORY TRACKING REPORT: _______-_______
(LOC-SN)

FRACTION CODE	X	PREP/ANAL REQUIRED	RESPONSIBLE INDIVIDUAL	DATE DELIVERED	DATE COMPLETED

CHAIN-OF-CUSTODY RECORD

G-30

CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME					NO. OF CON-TAINERS	ANALYSIS							REMARKS
SAMPLERS: *(Signature)*														
STA. NO.	DATE	TIME	COMP	GRAB	STATION LOCATION									

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

STATION LOCATION LOG

DATE: ______________

PROJECT: ____

STATION LOCATION: ______________________________________

DESCRIPTION OF SAMPLES COLLECTED: ____________________________

SPC ZONE: _________(N/S) EAST: __________ NORTH: __________

LOCATION:

Bottom Depth: _____ (ft) ________ (m) Tide: ± ______ (m) MLLW: _____ (ft) ______ (m)

LORAN C: LOP1 ______________ LOP2 ______________

Variable Radar Range: ______________________________________

Visual Fixes: (Note: Please tape any drawings to back of this sheet)

Photos - Roll: _______ Pictures: __________

PID Reading (range): ______________________________________

Comments: ____

RECORDER: _______ SIGNATURE: ____________ ORG. CORE ____ DATE: ____________